



# NORTH CAROLINA STATE BUREAU OF INVESTIGATION

## CRIME LABORATORY DIVISION

### TRACE EVIDENCE SECTION



## PROCEDURE FOR INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

### A. INSTRUMENTATION

The ICP-MS instrumentation currently in use consists of the following:

Perkin Elmer SCIEX Elan DRC-e Inductively Coupled Plasma Mass Spectrometer  
Perkin Elmer AS 93 Autosampler  
Polyscience Recirculator, Model 3370  
Elan ICP-MS Instrument Control Software, Version 3.0  
Dell Pentium 4 Processor w/ Microsoft Windows XP Professional  
Ultra High Purity Argon, Refrigerated Liquid

### B. PURPOSE

This method is designed to analyze primer residue for the presence of barium, antimony, and lead collected on the cotton swabs in the gunshot residue kits.

### C. GSR STANDARD AND SAMPLE PREPARATION

1. Sample Preparation - Prepare samples into 15 mL flacon tubes according to the Procedure for Gunshot Residue (GSR) Analysis and load them into the autosampler tray, beginning with autosampler position #38. \*The maximum sample load for the GSR analysis is 80 tubes or 16 GSR Kits.
2. Calibration Standards - Place Blank and Standards (prepared with the samples) in position #14 through #20. Place QC1 Solution in position #6. Place QC2 Solution in position #7.
3. Dual Detector Stock Solution - Add 0.2 mL of each 1000 ppm solution of Ba, Sb, Pb, In, and Lu to a 100 mL volumetric flask. Fill to volume with 1% Nitric Acid. Prepare a 1:10 dilution of stock to make a 200 ppm Ba, Sb, Pb, In, Lu for Dual Detector Calibration.
4. AutoLens Voltage Optimization- Elan 6100 Detection Limit Solution (10 ppb Be, Co, In, U)

## D. ICP-MS OPERATING PROCEDURES

1. Startup Procedures for ICP-MS and Software -p.2
2. Daily Performance (perform daily) -p.4
3. Optimizing the AutoLens Voltage -p.6
4. Calibrating the Dual Detector -p.6
5. Optimization Flowcharts -p.7-8.
6. Tuning Mass Spectrometer (perform when needed) -p.9
7. Setting up ICP-MS Software for GSR Analysis -p.9
8. Begin GSR Analysis -p.11
9. Saving Data to EXCEL file spreadsheet -p.11
10. Shutdown Procedure -p.12

## E. SAFETY CONCERNS

Any questions relating to safety are addressed in the ELAN ICP-MS Series Safety Manual 016628.

## F. OTHER INFORMATION

GSR Method - See Attachment

### Startup Procedures for ICP-MS and Software

\*\* This startup procedure is for normal daily activity. If the instrument is completely off, refer to the Hardware manual. The Order for switching the circuit breakers is:  
CB2 → CB4 → CB3 → CB1 → Vacuum ON

1. Check level of argon. The regulator should be set to at approximately 70psi. Check the second argon regulator connected to the inlet on the back of the ICP-MS. This pressure should be set at approximately 53psi.
2. Turn on the recirculator. The pressure should be set at approximately 50psi.
3. Attach the sample tubing and drain tubing leading from the peristaltic pump to the spray chamber. Remember when the pump turns on, it will rotate *counter-clockwise*. Be sure to follow the flow through the tubing as if the pump was on.

The flow direction for the sampling tubing (the smaller tubing) is from the sampling probe into the spray chamber.

The flow direction for the drain tubing (the thicker tubing) is from the bottom of the spray chamber / nebulizer to the waste collection bottle.

4. Attach the autosampler tubing in the same manner. Be sure to determine the direction of flow in the tubing if the pump turns *counter-clockwise*.

The flow direction for the rinse tubing (the smaller tubing) is from the 2 L rinse bottle (1 % HNO<sub>3</sub>) to the autosampler rinse position.

The flow direction for the drain tubing (the thicker tubing) is from autosampler probe rinse to the waste collection bottle.

5. Fill the 2 L rinse bottle with enough 1 % HNO<sub>3</sub> to complete anticipated run. Prepare 1 % HNO<sub>3</sub> according to Standard Operating Procedure For Gunshot Residue (GSR) Analysis.
6. Click on the ELAN icon on the computer desktop.
7. From the **File** menu, click on **Open Workspace...**
8. Select **Daily Performance.wrk** and click **Open**.
9. Turn on the autosampler by turning the power switch of the AUTOSAMPLER powerstrip to ON. The probe will run through a series of start up motions and come to rest above the probe rinse reservoir.  
  
*NOTE: Do not run samples using the autosampler until the probe rinse reservoir fills to the top and slowly drips to waste.*
10. Click on **Method** from the ELAN toolbar. (This is the icon toolbar across the top of the software window.) Click on the **Sampling** tab. Click on **Probe...** Click on **Goto Rinse**. This will place the probe into the 1% HNO<sub>3</sub> rinse.
11. Click on **Instrument** from the ELAN toolbar. Click on the **Front Panel** tab to determine the status of the instrument. The system status will display **READY** in green if all the hardware is operating properly and the plasma can be ignited. If the system status displays **NOT READY** in red, there is a fault in the system. Refer to **Diagnostic** and **Faults** tabs to determine the possible cause of the fault.
12. Click **START** under Plasma if the system is **READY**.
13. When the plasma is lit, all system parts will light up green and system status will read **READY** again.
14. Check flow of rinse as it begins to flow through the tubing.
15. Check the base vacuum pressure. For successful operation the pressure should read 10<sup>-6</sup> torr.

#### Daily Performance (perform daily)

\*\* A Daily Performance Check ICP-MS will be completed before the use of the instrument. A printed copy of each "Daily Performance Report" will be placed in the Daily Performance Log Book.

Procedures can be found in the Trace Evidence Section Calibrations Manual.

#### Tuning Mass Spectrometer (perform when needed)

\*\*Tuning is not normally a daily procedure. Tuning should be performed when there are changes to the instrument's electronics, whenever an elements resolution needs to be modified, or when performing a Complete

Optimization.

Procedures can be found in the Trace Evidence Section Calibrations Manual.

#### Setting up ICP-MS Software for GSR run

**\*\*Do not start a GSR run without running a Daily Performance Check.**

1. From the File menu, click on Open Workspace....
2. Select GSR.wrk and click OK.
3. Click on the Method icon on the Elan Toolbar.
4. Click on the Report Tab (located on the right-hand side of the Method window).
5. If a printout is desired after each run, under Report View, select Send to Printer by clicking on its box. For general quantitations and GSR analysis, select the following Report Option Template:  
  
C:\elandata\ReportOptions\quant summary1.rop
6. To create data files for each sample, under Report to File, select Send to File by clicking on its box. Select the following Report Option Template:  
  
C:\elandata\ReportOptions\exportdata.rop
7. Create a Report Filename specific for the run. This will be the name of the .csv file to later be converted into an EXCEL spreadsheet. Type the following:  
  
C:\elandata\ReportOutput\YOUR FILENAME GOES HERE.csv
8. Create a new Dataset for each GSR run. Select the Dataset icon from the Elan toolbar. From the File menu, select New. Type the desired dataset name then click on Open.
9. Create a new sample file for each GSR run. Select the Sample icon from the Elan toolbar. From the File menu, select Open.... Select the GSR-template .sam then click on Open.
10. Fill out the following information for each sample:  
  
Batch ID = Optional; can be used to distinguish multiple users in a single batch  
Sample ID = type the filename desired for each sample (SMITH C)  
Description = type the SBI Laboratory Case # here
11. Check the following information is correct:  
  
A/S Location = always begin sample in autosampler location 38; maximum of 85 samples  
Measurement Action = for first sample, select: Run Blank Std. and Sample  
= all following samples, select: Run Sample  
Method = C:\elandata\Method\GSR.mth

Sample Type = Sample  
Aliquot Volume = 1  
Diluted to Volume = 10

12. Check the following values are correct:

Sample Flush = 35  
Sample Flush Speed = -24  
Read Delay = 20  
Delay and Analysis Speed = -20  
Wash = 45  
Wash Speed = -24

**\*\*NOTE:** These value must be the same as the value entered in the Method for both Standards and QC.

13. The unused rows in the template must be deleted, when not performing a full 80 sample analysis. Left click on the Batch Index of the first unused row and drag down to the last Batch Index (usually 80). The unused rows should now be highlighted. Right click and select Delete.
14. Save the new sample file with a new name. From the File menu, select Save As... and type the new file name. Click Save.

#### Begin GSR Analysis

1. Click on Batch Index Number of the first sample and drag down until all samples for analysis are highlighted.
2. Click on Build Run List. (If a warning window pops up and says," Do you want to clear previously acquired QC Data before proceeding with the batch?" Click Yes.)
3. When the Run List Window opens, click Printable View.
4. Click on the printer icon to print the run list. Check to make sure the sequence of samples is correct.
5. Close this window.
6. Click on Analyze Batch. The run will start.

#### Saving Collected Data to EXCEL file spreadsheet

1. In WINDOWS, right click on the START button and select Explore.
2. Click on the folder: C:\Elandata\ReportOutput
3. Click on the appropriate .csv file for the GSR run. This will open the file into an EXCEL spreadsheet.

4. Highlight all of the data in the .csv file.
5. Go to the Edit menu and select Copy.
6. From the File menu select Open. Select the file: C:\GSR Casework\GSR-ICP-MS-template.xls
7. Click on Enable Macros.
8. Click on the A1 cell which reads "Paste Data Here".
9. Go to the Edit menu and select Paste.
10. Save this file under a new name.
11. Select all tabs for the kits that were run and print.
12. Each analyst will then move all dataset, sample, .csv, and excel files from the general locations to their personal folders.

#### Shutdown Procedure

1. Place the autosampler probe a bottle of deionized water and run through the system for ~5-10 minutes. Remove the probe and let pump run until all liquid has drained from the tubing into the waste.
2. Click on Instrument from the ELAN toolbar. Click on the Front Panel tab to determine the status of the instrument. Click STOP under Plasma. This will shut the plasma off and stop the peristaltic pump.
3. Flip the power to off on the autosampler powerstrip.
4. Turn off the recirculator.
5. Release the tension on the tubings from the peristaltic pump and the autosampler pump. This is the end of basic shutdown procedures. If there is a need to completely shutdown the system for an extended period of time, flip the power switches on the left side of the ICP-MS in the following order:

Vacuum Off → CB1 → CB3 → CB4 → CB2